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Procedia Engineering 10 (2011) 2147–2152

Engineering Procedia

ICM11

Precise Analysis of Near Surface Neutron Strain Imaging Measurements

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Abstract

The lateral resolution of Neutron Strain Imaging Instrumentation for high resolution applications is restricted. A realistic lower limit for reasonably fast measurements is 0.5 mm. The reason is, that in order to achieve maximum neutron flux at the sample position, the beam must be focussed. This is often achieved by double focussing monochromators or focussing guides. As a result the beam-divergence sets a lower limit for gauge volume definition.

However, the lateral resolution can be improved for near surface and interface analysis by entering the gauge volume only partially into the material. As a result, the sampled gauge volume is getting smaller and resolution is improved. As a drawback, errors of the detected peak position, usually called surface errors or pseudo-strain, are introduced in the measurements. Such errors are often corrected by repeating the measurement with the sample turned by 180°. This is inefficient, limited in precision and often not possible.

This paper describes a more efficient and precise method for near surface stress analysis that requires only one reference measurement, which provides experimental parameters for an analytical model for data correction. The model takes into account curved surfaces and allows precise stress determination as close as 40 micrometres below a surface or interface.

Pseudo peak shift ; radial focussing collimator ; near surface stress ; Neutron Strain Imaging

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 doi:10.1016/j.proeng.2011.04.355

1. Introduction

Neutron strain imaging is a powerful technique for the non-destructive three dimensional mapping of (residual) stress deep within matter. The lateral resolution typically lies in the millimeter and sub-millimeter range. The principle is that a gauge volume is defined by suitable beam optics and the diffraction peak from this volume is recorded. The peak position is referred to a reference and strain calculated from which stresses can be determined. By scanning the sample through the gauge volume, a strain/stress map can be produced. Since neutrons can penetrate a considerable amount of matter, neutron diffraction is used successfully for large specimens. In order to give an idea of possible penetration with neutrons examples are 60 mm thick steel or 300 mm Aluminium. Due to the relatively low neutron flux, compared to the photon flux at synchrotron x-ray beam lines, it is often not feasible to work with gauge volumes smaller than a few mm^3. One difficulty for the experimental set-up is that, in order to increase the efficiency, a divergent beam must be accepted. Therefore it is difficult to define a precise gauge volume. As a result, the method is said to be a low resolution technique for large samples.

However, the instrument SALSA [1] (Stress Analyser for Large scaled Applications) at the ILL (Institut Laue Langevin) in Grenoble, France, is capable to determine stresses from the bulk to about 40 μm to the surface. This is not only possible due to the use of radial focusing collimators, which provide precise beam-definition and allow divergent beams, but as well thanks to analysis software for the correction of experimental errors. The application and the background of this analysis software, developed by the author, is subject of this paper.

2. Experimental difficulties: Origin of Pseudo Peak Shift

As mentioned in the introduction, the generation of a precisely defined gauge volume is not evident on neutron instrumentation. In order to have sufficient neutron flux at the sample position, divergent beams are utilized, as delivered from focusing monochromators and super-mirror guides, for example. A gauge volume is defined in the centre of the instrument by using slit apertures or radial focusing collimators. Radial focusing collimators perform much better than slits in this case, since they transmit a divergent beam and at the same time define a precise beam profile [3]. But when beam dimensions much smaller than 1 mm are required, as for measurements of strain gradients in near surface regions, collimator manufacture comes to its technical limits. A way out of this is to scan the sample surface across the gauge volume. In this case the sampled gauge volume is smaller than the instrumental one and lateral resolution is improved. (The instrumental gauge volume is the gauge, defined by the beam shaping optics. The sampled gauge volume is the intersection between the instrumental gauge volume and the sample) The draw-back is that in this case additional peak shift is introduced to the recorded peak position. The origin of these pseudo peak shifts will be described in the following paragraph.

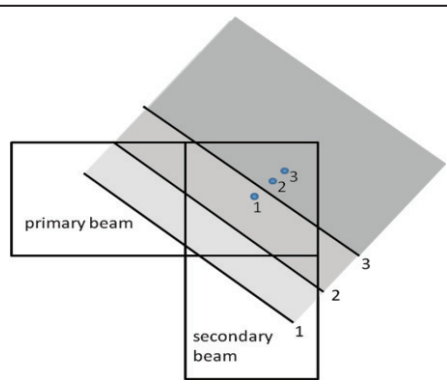

Fig. 1 centre of gravity positions of the sampled gauge volume for different sample positions during a through surface scan. Since their position is not in the middle of the gauge volume a geometric peak shift is detected.

A 2-axis diffractometer, this is what a strain imager actually is, is calibrated with respect to the instrument centre. This is at the same time the centre of rotation of the omega-table and the detector and the point to which the beam shaping optics are focusing the beam. If the sampled gauge volume is not centered over this point, obviously, the centre of gravity of the scattered intensity

is shifted (fig. 1). Now, if Position Sensitive Detectors are used, this translation is projected onto the detector and then interpreted as peak shift. This effect is called geometrical peak shift.

A second error source is present at instruments with a crystal monochromator. In order to gain intensity, monochromatic instruments use mosaic or bent crystal monochromators [2]. As a result the primary beam is not only divergent, but as well a wavelength-gradient is created in the primary beam. This gradient is such, that the diffracted Bragg-peak is focused on the detector. This improves the peak width and therewith strain resolution [2]. When now the sample material is only partially filling the gauge volume, the mean wavelength is different from the mean wavelength "seen" by the material when the gauge volume is fully embedded in the sample. As a result an additional peak shift is introduced. This shift can either add a huge amount on the geometrical or even compensate for it [4]. On instruments using a bent crystal monochromator this effect can be tuned. But it is then compromising peak width and intensity and the settings depend on the actual take-off angle and monochromator angle (=wavelength). In other words settings have to be found for each instrument configuration and their optimization is very time consuming. Alternatively the gradient can be measured and been taken into account by analysis software. This is described in the next section.

3. Determination of Pseudo Peak Shift

Since the displacement of the centre of gravity of the scattered intensity is moving in opposite directions when scanning either from the front-side or the back-side of a sample, the pseudo peak shift is moving in a point symmetric way (fig. 2). This can be used to determine this shift experimentally by performing a through surface scan a second time after rotation of the sample by 180°. The difference of the evolution of both measurements is the pseudo peak shift as a function of the sample position. In the same way, calculating the average between both curves gives the strain gradient in the sample, since the pseudo strain cancel out. This is a simple way of cancelling out experimental errors by just scanning a surface twice in two orientations. The disadvantage is that two measurements are required for each strain direction, which doubles the beam time. Furthermore such measurements are not possible, if the geometry of the sample does not allow transmission of the beam one of the orientations. This is often the case for the normal strain direction, since the beam must traverse the whole sample thickness. At 90° scattering geometry this is twice the path length compared to transmission geometry.

A more efficient solution is a mathematical description that can be fitted to the measurement and subtract the pseudo peak shifts; preferably without the need of a reference measurement for each single experiment. Such a model has been developed by the author. It is an analytical description of the peak shift, depending on sample position (within the gauge volume), taking into account scattering angle, beam intensity profile, linear absorption coefficient of the sample material and the wavelength distribution in the primary beam. The advantage is that, once the beam profile and the wavelength distribution are determined, the model can be used to analyze measurements at any diffraction angle 2Θ angle and for different materials.

The procedure is like this: An idealized reference sample – at SALSA a 4.6 mm thick polished parallel steel plate is used - in reflection and transmission geometry and as well in the 180° rotated orientations is measured. The pseudo peak shift is determined "experimentally" by subtracting the entrance and exit curve as described above. Then the mathematical model is applied, refining beam dimensions (primary and secondary), and wavelength distribution. These are the common parameters for every measurement, done at this wavelength-setting of the instrument. Only after wavelength change, this reference

measurement must be repeated, since the wavelength distribution will change too. Fig. 2 shows the pure geometric pseudo peak shift and the peak shift, taking into account wavelength gradient, which fits the experimental data very well.

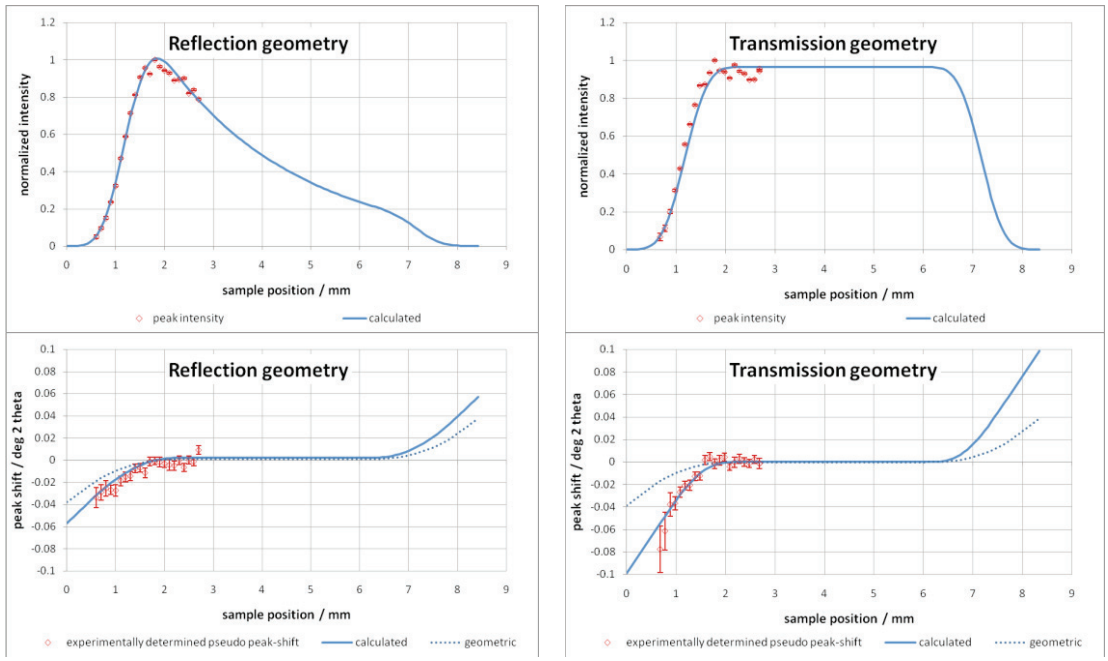

Fig. 2 Experimentally determined pseudo peak shift and analytical model, showing geometric shift (dotted line) and taking into account the wavelength distribution in the beam (solid line). On the left hand side in reflection geometry (i.e. normal strain component) and on the right in transmission geometry (i.e. in-plane component). On the top, the according intensity profiles, which allow precise determination of the sample position with respect to the gauge volume.

4. Analysis of measurements through flat surfaces or interfaces

The procedure for analyzing through surface scans is such that first the recorded intensity profile is fitted by the program. Beam dimensions and profile as well as wavelength gradient had been determined already from the instrument calibration measurement. The scattering geometry is given by the experimental set-up (detector angle). The parameters for the intensity fit are surface position and linear absorption coefficient of the sample material. (The upper graphs in Fig. 2 show a typical intensity profile in reflection and transmission geometry) The program then calculates for each measuring position the intersection between instrumental gauge volume and sample – the sampled gauge volume. The centre of gravity of the intensity distribution within this sampled gauge volume is taken as the co-ordinate of the "real" measuring point. A precision of <10 micrometres is possible, even with gauge volumes of 1 mm diagonal. As a basis for this calculation the beam intensity profiles are described as triangular shaped for radial collimators and trapezoidal for slits. For each real measuring point, the geometric pseudo peak shift and the peak shift due to the wavelength gradient are calculated and subtracted from the detected peak position.

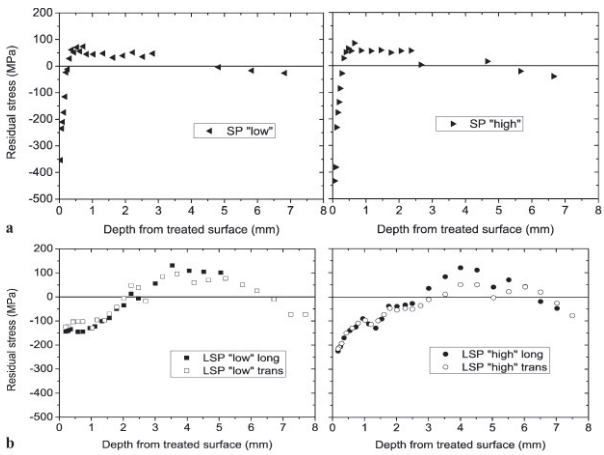

Fig. 3 shot peened (SP) and Laser Shock Peened (LSP) steel surfaces, measured using Neutron Strain Imaging (NSI) on SALSA. The results show, that LSP produces a far deeper compressive zone than SP. The graphs demonstrate that NSI is capable to resolve very near surface stresses in thick samples. Taken from [5]

An example of an application, measured at SALSA is a comparative study of surface treatments for improving fatigue behavior of components [5]. Fig. 3 shows results of stress measurements in 8 mm thick type 304 austenitic steel plates, treated by shot peening (SP) and Laser Shock Peening (LSP). It is advantageous of using relatively thick substrate materials for investigations of peening induced stresses, since the deformation of the substrate - due to the introduced stresses – is then reduced. Otherwise stress levels from peening would be reduced. Neutron Strain Imaging is capable to perform complete stress analysis and resolves stresses even 30 micrometres from the surface, as shown from the LS-measurement in fig. 3.

5. Measurements at curved surfaces

Since the intensity evolution is used to determine the precise position of each measuring point, it must be described very precisely by the model. When a scan is performed through a curved surface, the path-length of each neutron beam traversing the sample is not the same as for a sample with parallel plane surfaces. However, the above described model can be used for fitting, if the intensity plot is transformed. This is done by transforming the co-ordinate of each measuring point in the cylindrical sample to a co-ordinate at which the intensity is the same as in a plane sample. Then the plane-model can be used for fitting and pseudo peak shift correction. Afterwards the co-ordinates of measuring positions are transformed back.

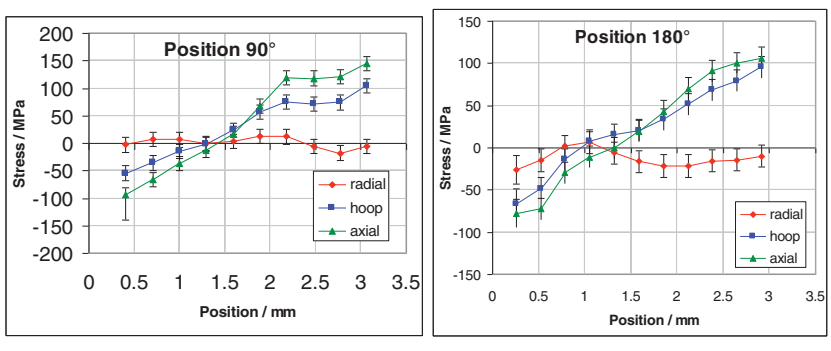

Fig.4 Stress gradient within the 3.2 mm thick wall of a drawn Copper tube at two different positions around the circumference where the material has seen different deformation. From [5]

An example where this technique has been applied is shown in fig. 4. It shows the stress profile in walls of a precision drawn SF-Copper tube [6]. The tube had awall thicknesse of 3.4 mm and shows a remarkable stress gradient across thickness. Since the interest of the investigation was not the determination of a near surface stress gradient, but the detection of the gradient inside the wall, the step width of the experiment was chosen to 0.3 mm. Since the diagonal of the gauge

volume had a length of 1.9 mm, the first approximately 1.2 mm either side (2/3 of the gauge volume diagonal), are affected by pseudo peak shifts. Only one millimeter in the inside is not affected by pseudo peak shifts. By applying the above described analysis model for pseudo peak shift correction, all data points could be analyzed, showing the complete stress gradient and the stress level near the surfaces.

6. Summary

It has been demonstrated that Neutron Strain Imaging is not only capable to map stresses in thick materials, but can as well be used for high resolution measurements for near surface or interface stresses. Pseudo peak shifts are introduced by the experimental set-up which have their origin in a geometric displacement of the centre of gravity of the intensity distribution in the sampled gauge volume plus the wavelength-distribution present in the primary beam. An analytical mathematical model has been developed which requires only one reference measurement for the determination of instrumental parameters and can then correct measurements for pseudo peak shift and determine the measuring positions.

The features of the model can be summarised as follows:

- Only one reference measurement is required per wavelength setting
- The sample for the reference measurement can be idealized in terms of geometry, dimensions and material – so the reference measurement is fast.
- The reference measurement does not need to be performed at the scattering geometry of the actual experiment. (=> choice of material)
- Even data-sets can be analyzed, where only few data points are recorded in near surface regions.
- The model describes flat and cylindrical sample shapes, such as tubes

The model is used regularly on SALSA for the analysis of near surface stress gradients and allows precise measurements as close as 40 micrometres from a surface or interface. The model allows as well analysis of measurements in thin walled samples. A practical limit on SALSA is 0.5 mm.

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